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(54) Title: CORROSION INHIBITOR AND PROCESS

(57) Abstract

A corrosion inhibitor composition for metal working and a method for making the same is provided. The composition is made by reacting maleated tall oil fatty acids with alkanolamines. The alkanolamines suitable for the present invention may be ethanolamines or other alkanolamines such as di–glycolamine if ethanolamines are not environmentally preferred. The resulting reaction product salts provide an effective corrosion inhibitor suitable for incorporation into aqueous products such as buffing compounds, cutting fluids, drawing compounds, cleaning fluids and forging aids.

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CORROSION INHIBITOR AND PROCESS

Field of the Invention

The present invention is generally directed to a corrosion inhibitor and a process for using the inhibitor. More particularly, the present invention is directed to a corrosion inhibitor particularly well suited for preventing corrosion during various metal working operations.

Background of the Invention

This invention relates to corrosion inhibitors used in metal working. As used herein, metal working includes, but is not limited to, metal polishing, cutting, grinding, stamping, cleaning, drawing, forming, forging, and other processes where metal oxidation and corrosion is a problem.

It is well known in the metal working industries to combine corrosion inhibitors with cutting fluids, cleaners, and drawing compounds. Traditionally, typical inhibitors employ alkyl diacids of C_6 to C_{12} chain length such as dodecanedioic acid to protect the metal.

U.S. Patent No. 5,292,480 to Fischer, et al. Discloses a corrosion inhibitor for metal parts which is produced by reacting a fatty acid Diels-Alder adduct or fatty acid-ene with a polyalcohol to form an acid-anhydride ester corrosion inhibitor. The resulting ester may be further reacted with amines, metal hydroxides, metal oxides, ammonia, and combinations thereof to neutralize the ester. The corrosion inhibitor is disclosed as having utility for controlling corrosion in down-hole oil field and piping equipment.

U.S. Patent No. 5,643,534 to Minevski

discloses a corrosion inhibiting composition supplied as a concentrate which includes a blend of salicylic acid; a reaction product of maleated tall oil fatty acid polyhydroxy polyalkane and ammonium or potassium hydroxide; tallow diamine quaternary dichloride or alkanediol; and alkanol or alkanolamine. The inhibitor is disclosed as being useful in acid gas removal amine units which employ alkanol amine solutions.

While these corrosion inhibitors are believed satisfactory for their intended purposes, there exists room for improvement within the art. The present invention is an improvement in that it provides an effective corrosion inhibitor which is cost effective, easy to manufacture, and affords superior corrosion protection. A preferred embodiment of the composition has been found useful as an aqueous-based corrosion inhibitor which is compatible with a variety of metal working formulations and processes.

Summary of the Invention

It is an object of the present invention to provide a composition useful as a corrosion inhibitor for metal working applications.

It is another object of the present invention to provide a corrosion inhibitor composition which is compatible with conventional additives used in the metal working industry.

It is a further and more particular object of this invention to provide a corrosion inhibitor with a broad range of utility and which has low toxicity.

It is a further object of this invention to provide a process of preventing corrosion in worked metals which comprises the use of a reaction

product of a maleated tall oil fatty acid with one or more alkanolamines.

These and other objects of the present invention are achieved by providing a metal corrosion inhibitor composition. The corrosion inhibitor composition includes a reaction product of a maleated tall oil fatty acid with an alkanolamine. The alkanolamine can be, for instance, an ethanolamine, such as monoethanolamine, triethanolamine, and mixtures thereof. When in use, the corrosion inhibitor composition of the present invention can be contained in a dilute aqueous solution. For instance, the corrosion inhibitor composition can be present in the solution in an amount up to about 20% by weight, and particularly in an amount up to about 5% by weight.

In one embodiment of the present invention, the corrosion inhibitor composition can further include a reaction product of an alkanolamine and a C_6 to C_{18} alkyl carboxylic acid. The C_6 to C_{18} alkyl carboxylic acid can be, for example, neodecanoic acid, isononanoic acid, and mixtures thereof.

The metal corrosion inhibitor composition of the present invention is well suited to protecting metals when the metals are contacted with fluids, such as water, that can cause corrosion. For example, in one embodiment, the corrosion inhibitor of the present invention can be incorporated into a lubricating solution that lubricates the metal during metal working operations. The corrosion inhibitor composition can also be incorporated into rinsing solutions.

Description of Preferred Embodiments

It is to be understood by one of ordinary

skill in the art that the present discussion is a description of exemplary embodiments only, and is not intended as limiting the broader aspects of the present invention, which broader aspects are embodied in the exemplary construction.

A composition and process for treating metals, especially ferrous metals, to prevent corrosion are provided. Generally, the composition comprises a reaction mixture of a maleated tall oil fatty acid with one or more alkanolamines. Maleated tall oil fatty acid is known as an intermediate chemical for various corrosion inhibitors. However, the specific reaction products of the present invention offer improved qualities over other known derivatives of maleated tall oil fatty acid. The resulting reaction products of the present invention comprise alkanolamine salts of the maleated tall oil fatty acid and afford excellent corrosion resistance to worked metals. The reaction product(s)are water soluble; are compatible with typical metal working additives commonly used in metal finishing, buffing compounds, cutting fluids, and other metal working compounds; and have low toxicity.

An additional advantage of the present compositions and process is the ease of manufacture. The present inhibitor formulation is made according to a process which takes place in simple reactor vessels at ambient laboratory conditions and pressures. While the reaction is slightly exothermic, no external pressure or temperature regulation is required. Further, the reaction proceeds quickly, in contrast to conventional teachings where elevated pressures,

temperatures and/or reaction times are needed.

As described above, in general, the corrosion inhibitor composition of the present invention includes the reaction products of a maleated tall oil fatty acid with one or more alkanolamines. Alkanolamines that may be used in the present invention can vary depending upon the particular application and the particular result desired. For most applications, ethanolamines can be used to neutralize the maleated tall oil fatty acid. Particular ethanolamines that can be used include triethanolamine and monoethanolamine. In fact, it has been discovered that when a mixture of triethanolamine and monoethanolamine are used to neutralize the tall oil fatty acid, superior results are achieved. For instance, a triethanolamine and monoethanolamine mixture can be used to neutralize the fatty acid in which triethanolamine is present in the mixture in an amount of at least 30% by weight, particularly at least 50% by weight and, in one embodiment, present in an amount of about 66% by weight.

In general, one or more alkanolamines are combined with a maleated tall oil fatty acid in an amount sufficient to completely neutralize the fatty acid. Preferably, excess amounts of alkanolamines are present. For most applications, the weight ratio of alkanolamines to the maleated tall oil fatty acid is about 2 to 1.

Besides at least one alkanolamine and the maleated tall oil fatty acid, the reaction mixture of the present invention can include various other ingredients. For instance, various additives can be included in order to facilitate the reaction or in order to enhance the final properties of the

composition. For example, in one embodiment, one or more alkyl carboxylic acids having from a C_6 to a C_{18} carbon chain may be added to the reaction mixture. Specifically, the alkyl carboxylic acid can be added in order to improve the properties of the final product. During the reaction, the alkyl carboxylic acid also becomes neutralized by the alkanolamines.

Examples of alkyl carboxylic acids that may be used in the process of the present invention include neodecanoic acid and isonononoic acid.

Once reacted, the corrosion inhibitor composition of the present invention should generally have a pH of greater than about 6, and preferably has a pH of greater than about 7. In one embodiment, the composition can have a pH of from about 7 to about 9. It should be understood, however, that higher pHs are possible.

When used in metal working applications, the reaction products of the present invention can be combined in an aqueous solution. For most applications, the corrosion inhibitor composition of the present invention can be heavily diluted with water during use. For instance, the concentration of the reactant products in an aqueous solution during use is typically from about 0.25% to about 1% by weight.

The corrosion inhibitor of the present invention has been found to be compatible with a variety of standard additives found in metal working applications. For instance, the corrosion inhibitor composition can be combined with other corrosion inhibitors, surfactants, and lubricants.

For instance, in one embodiment of the present invention, the corrosion inhibitor composition can

be contained within a lubricating solution that is intended to treat metal substrates when the substrates are subjected to various metal working processes. Examples of surfactants that may be used with the composition of the present invention include, but are not limited to, fatty acid alkoxylates, fatty amine alkoxylates, alcohol alkoxylates, alkylphenol alkoxylates, castor oil alkoxylates, phosphate esters and polyols. Further, it is believed that any ethoxylated or propoxylated derivative may be combined and used with the composition of the present invention.

Metal lubricants that may be used with the composition of the present invention include most synthetic or nonsynthetic lubricants. Examples of lubricants include, for instance, mineral oil, vegetable oil, fatty esters, polyol esters, and any of the above described surfactants, which sometimes also serve as lubricants. In general, when treating metals, a lubricant solution can contain a lubricant in an amount up to about 80% by weight, particularly in an amount up to about 50% by weight, and more particularly in an amount from about 3% to about 40% by weight. The remainder of the solution can include the corrosion inhibitor composition of the present invention, any surfactants, and water.

When in use, a lubricating composition is typically applied to a metal while the metal is being subjected to some type of process, such as a forming, cutting, stamping, drawing, polishing, grinding, or forging process. After the metal has been worked, the lubricating solution containing the corrosion inhibitor composition of the present invention is typically washed off. Of particular

advantage, as described above, the corrosion inhibitor composition of the present invention is nontoxic and thus can be easily disposed of.

Besides being contained in a lubricating solution, the corrosion inhibitor composition of the present invention can also be contained in the rinsing solution and in other various solutions that contact the metal.

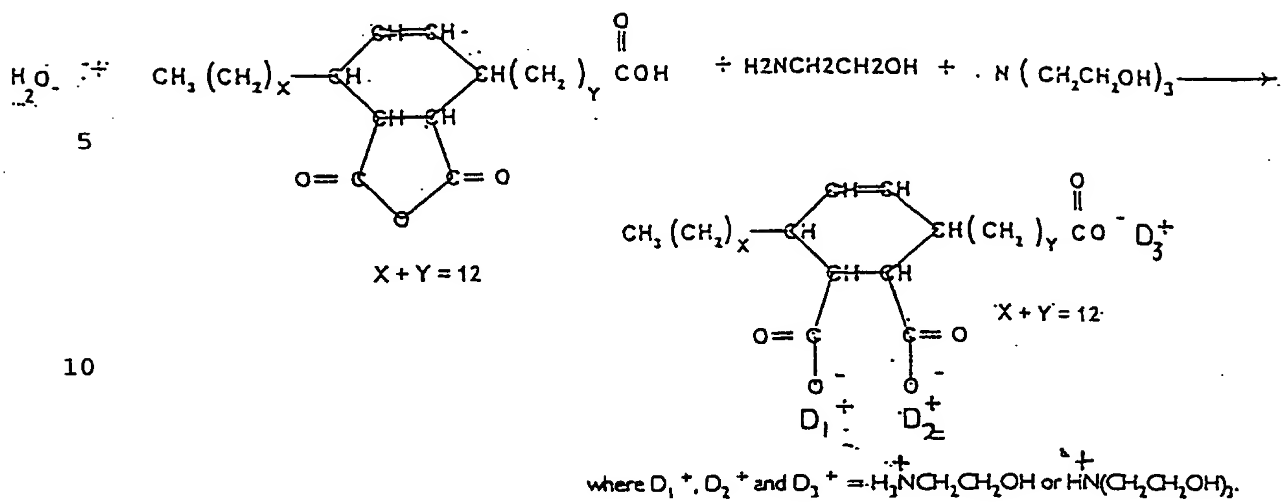
Example 1

A first preferred embodiment of the present invention is provided by the reaction of a maleated tall oil fatty acid (herein after "tall oil"), such as that available from Westvaco as TENAX 2010, with a mixture of alkanolamines, such as monoethanolamine (MEA) and triethanolamine (TEA). The maleated tall oil fatty acid used herein, has a monomer content of less than about 10%.

The reaction products are obtained as follows:

A 24.4% by weight of water is placed in a reaction vessel with stirring. 12.2% by weight of MEA (Dow; Union Carbide) and 37.8% by weight of TEA (Dow; Union Carbide) are added to the water with constant stirring or agitation. Once throughly mixed, 25.6% by weight of TENAX 2010 brand of tall oil is slowly added with constant stirring. The resulting exothermic reaction is allowed to proceed for approximately 30 minutes. A final pH of between 7-9 is obtained for the reaction product and may be adjusted using additional TEA or other similar acids.

The resulting reaction products are the amine salts of a polycarboxylic acid such as the tricarboxylic acid set forth below. The reaction products are in the form of a viscous, dark amber solution. The reaction and resulting reaction products are set forth in the following equation:



As set forth in Examples 2 and 3, additional carboxylic acids such as neodecanoic acid and isonononoic acid may be added/reacted along with the tall oil. The reaction products and formulations set forth herein, provide a stable, versatile, water soluble corrosion-inhibiting formulation which is compatible with a variety of additional additives, including surfactants, lubricants, and other inhibitors.

The reaction mixture's primary products are the salts referenced in the reaction products above. A small percentage of an ester amide reaction product may be present, but such products are not thought to significantly contribute to the corrosion inhibiting properties seen herein.

As set forth in the examples below, the combination of approximately 3 parts of TEA to 1

part of MEA has been found to perform best under the described test conditions. However, the use of TEA as the sole alkanolamine also provides a reaction product useful as a corrosion inhibitor. Beneficial results are achieved using diethanolamine (DEA) as the alkanolamine. However, as a suspected carcinogen, the use of DEA is not preferred. The formulations described above are preferred and achieve the beneficial results set forth in the additional examples below.

Example 2

A second preferred embodiment of the corrosion inhibitor formulation was prepared as described above, using the ingredients and proportions set forth in Table 1. The stock corrosion inhibitor as set forth in Table 1 was mixed in distilled water to provide working concentrations of 1%, 0.75%,0.5%, and 0.25%.

Each working concentration was used to evaluate the corrosion inhibiting properties with respect to cast iron chips in a open environment. Specifically, the above solutions were tested for their ability to inhibit corrosion according to standard test method ASTM-D4627-86. 10 grams of cast iron chips were placed in contact with a circular filter paper contained within a 9 cm petri dish bottom. 40 mls of the respective concentrations of the corrosion inhibitor were added to the petri dish, uniformly covering and wetting the cast iron chips. Air bubbles were displaced by gentle tapping of the petri dish, ensuring all the iron chips were in contact with the inhibitor solution.

Following an incubation period of 30 minutes, the inhibitor solution was decanted and the excess

free fluid was removed. The iron chips were left undisturbed for 24 hours in the open petri container and under standard ambient laboratory conditions. After 24 hours, the iron chips were removed from the filter paper and the number of rust spots appearing on the paper were recorded. The results are reported based upon the following rankings:

0-5 spots Excellent
5-10 spots Good
10-20 spots Fair
>20 spots Poor

Observations of the surface of the iron chips were also made. Further, qualitative assessments were possible by comparing the intensity of stains present on the filter paper.

TABLE 1

	1	2
H_2O	22.0	22.0
MEA	11.0	11.0
TEA	34.0	34.0
TENAX 2010	23.0	23.0
Neodecanoic Acid	10.0	--
Isonononoic Acid	--	10.0
Results of Cast Iron Chip Test at 1%	Excellent	Excellent
Results of Cast Iron Chip Test at 0.75%	Excellent	Excellent
Results of Cast Iron Chip Test at 0.5%	Excellent	Excellent
Results of Cast Iron Chip Test at 0.25%	Fair	Poor

All values are in wt. %.

As seen in reference to Table 1, the corrosion inhibitor provided excellent corrosion protection to the test iron particles.

Example 3

By way of comparison, Table 2 sets forth data on formulations where the maleated tall oil fatty acid was replaced with various carboxylic acids. The carboxylic acids evaluated are known and used for their corrosion inhibiting properties. In each instance, the listed formulations were evaluated using the chip iron protocol of Example 2 and the results reported in Table 2.

TABLE 2

	1	2	3	4	5	6
H_2O	35.0	35.0	30.0	30.0	20.0	20.0
TEA	34.0	34.0	34.0	34.0	52.0	52.0
MEA	11.0	11.0	11.0	11.0	5.0	5.0
Octadecenyl Succinic Acid Anhydride	10.0	10.0	15.0	15.0	--	--
D. Basic Acid	--	--	--	--	8.0	8.0
Azelaic Acid	--	--	--	--	15.0	--
Sebacic Acid	--	--	--	--	--	15.0
Neodecanoic Acid	10.0	--	10.0	--	--	--
Isonononoic Acid	--	10.0	--	10.0	--	--
Results of Cast Iron Chip Test at 1%	Poor	Poor	Fair	Poor	Poor	Poor
Results of Cast Iron Chip Test at 0.75%	Fail	Fail	Poor	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.5%	Fail	Fail	Fail	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.25%	Fail	Fail	Fail	Fail	Fail	Fail

All values are in wt. %.

Example 4

As set forth in Tables 3 and 4, various combinations of formulations were evaluated using TENAX 2010 in combination with other amino alcohols. In particular, in this example, the monoethanolamine and the triethanolamine were replaced with the amino alcohols. The protocols and testing procedures used in Example 2 were followed. Results are as reported in Tables 3 and 4.

TABLE 4

	1	2	3	4	5	6
H_2O	20.0	20.0	20.0	15.2	15.4	11.3
TENAX 2010	40.0	40.0	40.0	54.5	46.7	44.6
2 amino - 2 ethyl - 1,3 propanediol (85% conc.)	--	40.0	20.0	--	--	20.0
2 amino - 2 methyl - 1 propanol (95% conc.)	40.0	--	20.0	30.3	26.6	24.1
Neodecanoic Acid	--	--	--	--	11.3	--
Results of Cast Iron Chip Test at 1%	Fair	Poor	Poor	Poor	Poor	Poor
Results of Cast Iron Chip Test at 0.75%	Poor	Fail	Fail	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.5%	Fail	Fail	Fail	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.25%	Fail	Fail	Fail	Fail	Fail	Fail

All values are in wt. %.

TABLE 3

	1	2	3	4	5	6	7
H_2O	20.0	--	--	16.7	15.3	18.2	18.2
TENAX 2010	50.0	50.0	50.0	41.7	46.2	54.5	54.5
2 amino - 2 ethyl - 1,3 propanediol (85% conc.)	25.0	25.0	--	10.4	23.1	--	--
2 amino - 2 methyl - 1 propanol (95% conc.)	25.0	--	25.0	10.4	--	27.3	--
Monoisopropanol amine	--	25.0	25.0	20.8	7.7	--	27.3
Neodecanoic Acid	--	--	--	--	7.7	--	--
Results of Cast Iron Chip Test at 1%	Poor	Poor	Poor	Poor	Poor	Poor	Poor
Results of Cast Iron Chip Test at 0.75%	Fail	Fail	Fail	Fail	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.5%	Fail	Fail	Fail	Fail	Fail	Fail	Fail
Results of Cast Iron Chip Test at 0.25%	Fail	Fail	Fail	Fail	Fail	Fail	Fail

All values are in wt. %.

As set forth above, the preferred embodiment of the corrosion inhibitor affords significant corrosion protection to the test metals. Compositions replacing the tall oil with other carboxylic acids (Table 2) resulted in products which performed poorly compared to the tall oil reaction products of the present invention. Similar substitution efforts with respect to the preferred alkanolamines (Table 3 and 4), also failed to achieve results as good as the preferred compositions. The combination of MEA/TEA with the maleated tall oil produces a reaction product having superior performance qualities. In fact, a true synergy is achieved in the preferred combinations when compared to the corrosion inhibiting properties of the various reagents as evaluated in Tables 3 and 4.

The preferred embodiment of the present invention has demonstrated its effectiveness compared to other corrosion inhibitors evaluated. The corrosion inhibitors of the present invention offer greater protection than conventional inhibitors. Further, the present formulation achieves improved results while using lower concentrations than prior art formulations. Accordingly, the present formulations are advantageous in terms of economy of cost as well as effectiveness.

It will be understood that the invention is not limited to the particular embodiments or processes described herein, nor the particular parameters or dimensions described therefor. It should also be understood that other basic fatty acid material equivalent to that described falls within the scope of the present invention.

Preparation routes of the compositions and process steps therefore are merely exemplary so as to enable one of ordinary skill in the art to employ the method and compositions described herein and used in accordance with the present process. It will also be understood that while the form of the invention shown and described herein constitutes a preferred embodiment of the invention, this description is not intended to illustrate all possible forms of the invention. Aspects of the various embodiments may be interchanged both in whole or in part. The words used are words of description rather than of limitation. Various changes and variations may be made to the present invention without departing from the spirit and scope of the following claims.

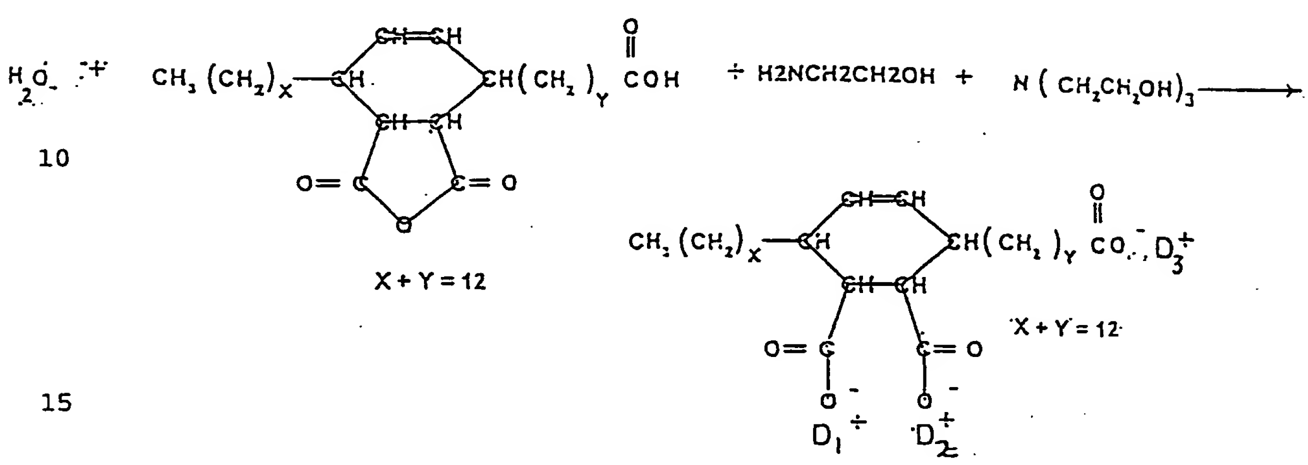
WHAT IS CLAIMED:

1. A metal corrosion inhibitor composition comprising:

a reaction product of a maleated tall oil fatty acid with an alkanolamine.

2. The composition as defined in claim 1, wherein said alkanolamine is an ethanolamine selected from the group consisting of monoethanolamine, triethanolamine, and mixtures thereof.

3. The composition as defined in claim 2, wherein said reaction products have a general chemical structure consisting of:



where D_1^+, D_2^+ and D_3^+ = $H_3\overset{+}{N}CH_2CH_2OH$ or $H\overset{+}{N}(CH_2CH_2OH)_3$.

4. The composition according to claim 1, wherein said maleated tall oil fatty acid and said alkanol-amine is reacted in the presence of neodecanoic acid.

5. The composition according to claim 1, wherein said maleated tall oil fatty acid and said alkanol-amine is reacted in the presence of isonononoic acid.

6. The composition according to claim 1, wherein said maleated tall oil fatty acid and said alkanol- amine is reacted in the presence of a mixture of isonononoic acid and neodecanoic acid.

7. The composition according to claim 2, wherein about 1 part of said maleated tall oil fatty acid is present to about 2 parts of said alkanolamines.

8. The composition according to claim 1, wherein said maleated tall oil fatty acid and said alkanolamine are reacted in the presence of a C_6 to C_{18} alkyl carboxylic acid.

9. The composition according to claim 1, wherein said composition comprises an aqueous solution.

10. The composition according to claim 2, wherein said alkanolamine comprises a mixture of monoethanolamine and triethanolamine, and wherein about 1 part of said monoethanolamine is present to about 3 parts of said triethanolamine.

11. The composition as defined in claim 1, wherein, said alkanolamine comprises a material selected from the group consisting of a glycolamine, tri-isopropanolamine, mono-isopropanolamine, and mixtures thereof.

12. A process of inhibiting corrosion of metals, said method comprising adding to a metal an effective amount of an aqueous solution containing a corrosion inhibiting composition, said composition comprising a reaction product of a maleated tall oil fatty acid and an alkanolamine.

13. The process as defined in claim 12, wherein said alkanolamine comprises at least one ethanolamine.

14. The process according to claim 13, wherein said ethanolamine is triethanolamine.

15. The process according to claim 13, wherein said ethanolamine is monoethanolamine.

16. The process according to claim 13, wherein said ethanolamine is a mixture of monoethanolamine and triethanolamine.

17. The process according to claim 16, wherein said mixture of monoethanolamine and triethanolamine contains at least 50% by weight triethanolamine.

18. A process as defined in claim 12, wherein said corrosion inhibiting composition further comprises a reaction product of an alkanolamine with a C_6 to C_{12} alkyl carboxylic acid.

19. The process according to claim 18, wherein said C_6 to C_{18} alkyl carboxylic acid comprises a material selected from the group consisting of neodecanoic acid, isonononoic acid and mixtures thereof.

20. The process according to claim 12, wherein said alkanolamine comprises a material selected from the group consisting of a glycolamine, tri-isopropanolamine, mono-

isopropanolamine, and mixtures thereof.

21. A process for working metals, said process comprising the steps of:

providing a metal substrate;

contacting said metal substrate with an aqueous solution, said aqueous solution containing a lubricant and a corrosion inhibitor, said corrosion inhibitor comprising a reaction product of a maleated tall oil fatty acid and an alkanolamine; and

working said treated metal substrate in order to modify the shape of said substrate.

22. A process as defined in claim 21, wherein said alkanolamine comprises at least one ethanolamine.

23. A process as defined in claim 22, wherein said ethanolamine comprises monoethanolamine.

24. A process as defined in claim 22, wherein said ethanolamine comprises triethanolamine.

25. A process as defined in claim 22, wherein said ethanolamine comprises a mixture of triethanolamine and monoethanolamine.

26. A process as defined in claim 25, wherein said mixture of said monoethanolamine and triethanolamine comprises at least 50% by weight triethanolamine.

27. A process as defined in claim 21, wherein said aqueous solution further comprises a surfactant.

28. A process as defined in claim 21, wherein said corrosion inhibitor further comprises a reaction product of an alkanolamine and a material selected from the group consisting of neodecanoic

acid, isononanoic acid, and mixtures thereof.

29. The process according to claim 21, wherein said alkanolamine comprises a material selected from the group consisting of a glycolamine, tri-isopropanolamine, mono-isopropanolamine, and mixtures thereof.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/05613

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :C23F 11/10
US CL :422/16,17; 252/392
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 422/7, 16, 17; 252/390, 393

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

STN: structure search and text search: tall oil fatty acid, maleic, maleated, amine

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- A	US 5,759,485 A (FISCHER ET AL) 02 June 1998 (02.06.98), see entire document.	1,9,12,13, 18,21,22, 27 --- 2-8,10,11, 14-17,19, 20,23-26, 28,29
A	EP 0711850 A1 (BOCKOWSKI ET AL) 15 May 1996 (15.05.96), see entire document.	1-29

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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